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(54) PRODUCTION OF POLYOXYALKYLENE AMIDE OF FATTY ACID

(57)Abstract:

PROBLEM TO BE SOLVED: To obtain a compound capable of being widely used for cosmetics and improved in stability during storage by removing low boiling components with water by distillation after carrying out an addition reaction of raw materials.

SOLUTION: This production of a polyoxyalkylene amide of a fatty acid is to carry out an addition reaction by adding a 2-4C alkyleneoxide to an alkanolamide of a fatty acid expressed by the formula, RCON(X)p(Y-OH)m [R is a 7-21C alkyl, etc.; X is H or CH3; Y is 1-5C alkylene; (p) is 0 or 1; (m) is 1 or 2]. For the addition reaction, it is preferable to use 0.01-5mol of a basic catalyst, e.g. NaOH, etc., based on 1mol of the compound of the formula and a reaction temperature is preferably equal to or more than the melting point of the compound of the formula and ≥110°C, especially 80-100°C is preferable. An added mol number of the alkyleneoxide is preferably 1-20mol in average and 1-10mol are more preferable. The obtained crude product is subjected to removement of components having low boiling points by distillation together with the catalyst and water. As the compound of the formula used, monoethanolamide is especially preferable.

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FULL CONTENTS

[Claim(s)]

[Claim 1] General formula (1)

RCON(X)p(Y-OH)m (1)

(As for the inside of a formula, and R, an example and X show the alkyl group or alkenyl group of the straight chain of carbon numbers 7-21, or branched chain, H or CH3, and Y show the alkylene machine of the straight chain of carbon numbers 1-5, or branched chain, and, as for 0 or 1, and m, p shows 1 or 2.) However, it is p+m=2. The manufacture method of the polyoxyalkylene fatty acid amide characterized by carrying out evaporation removal of the low-boiling point ingredient with moisture after making the alkylene oxide of carbon numbers 2-4 add to the fatty acid alkanolamide expressed.

[Claim 2] The manufacture method of the polyoxyalkylene fatty acid amide according to claim 1 which is the method which the method of carrying out evaporation removal of the low-boiling point ingredient with moisture shows to Following a and b.

a) How to perform by introducing steam or water the bottom of the condition more than **** of water, and in the system of reaction.

b) How to perform by making the inside of the system of reaction after introducing water in the system of reaction into the conditions more than **** of water.

[Claim 3] The manufacture method of polyoxyalkylene fatty acid amide according to claim 1 or 2 that the amount of moisture removed with a low-boiling point ingredient is 1 to 50 weight % to polyoxyalkylene fatty acid amide.

[Claim 4] The manufacture method of Claim 1 whose fatty acid alkanolamide expressed with a general formula (1) is fatty acid monoethanolamide or fatty acid diethanolamide - polyoxyalkylene fatty acid amide given in any 1 clause of three.

[Claim 5] The manufacture method of Claim 1 whose alkylene oxide is ethylene oxide and the number of average addition Mol of whose is 1-20mol - polyoxyalkylene fatty acid amide given

in any 1 clause of four.

[Detailed Description of the Invention]

[0001]

[Field of the Invention] About the manufacture method of polyoxyalkylene fatty acid amide, in detail, it gels at the time of preservation, or this invention relates to the manufacture method of polyoxyalkylene fatty acid amide excellent in the preservation stability which produces neither coloring nor muddiness.

[0002]

[Description of the Prior Art] Polyoxyalkylene fatty acid amide is widely used for a shampoo, kitchen detergent, cosmetics, etc. as the washing power improvement agent of a liquid detergent, a foaming agent, and bubble stabilizer. Moreover, by low stimulativeness, since the various derivatives obtained by performing carboxymethyl-izing, phosphoric-ester-izing, and sulfate ester-ization to this fatty acid amide further are also excellent in foamability and detergency, they are blended with the detergent constituent etc. as a main base of a surface-active agent etc.

[0003] As the manufacture method of this polyoxyalkylene fatty acid amide, The fatty acid alkanolamide obtained by the reaction of the low-grade ARUKIRU ester of fatty acid and fatty acid or the glycerin ester of fatty acid, and alkanol amine is used as materials. To this fatty acid alkanolamide, [alkylene oxide] The method of making it add is known (E.Jungermann). and D.Taber and Nonionic Surfactants (1) ed.by M.J.Schick, Marcel Dekker, Ink., New York, 1967, pp.211-222.

[0004] However, the polyoxyalkylene fatty acid amide obtained by this method had the fault which preservation stability is inferior in and produces muddiness with the serious fault of gelling at the time of preservation of coloring.

[0005] Therefore, the purpose of this invention is to offer the polyoxyalkylene fatty acid amide excellent in preservation stability.

[0006]

[Means for Solving the Problem] After this invention persons made alkylene oxide add to fatty acid alkanolamide as a result of examining this situation wholeheartedly, by carrying out evaporation removal of the low-boiling point ingredient with moisture, they find out that preservation stability is improved greatly and came to complete this invention.

[0007] That is, this invention is a general formula (1).

RCON(X)p(Y-OH)m (1)

(As for the inside of a formula, and R, an example and X show the alkyl group or alkenyl group

of the straight chain of carbon numbers 7-21, or branched chain, H or CH3, and Y show the alkylene machine of the straight chain of carbon numbers 1-5, or branched chain, and, as for 0 or 1, and m, p shows 1 or 2.) However, it is p+m=2. After making the alkylene oxide of carbon numbers 2-4 add to the fatty acid alkanolamide expressed, the manufacture method of the polyoxyalkylene fatty acid amide characterized by carrying out evaporation removal of the low-boiling point ingredient with moisture is offered.

[0008]

[Embodiment of the Invention] The form of operation of this invention is explained in detail hereafter.

[0009] it sets to the fatty acid alkanolamide expressed with the general formula (1) used by this invention -- although R shows the alkyl group or alkenyl group of the straight chain of carbon numbers 7-21, or branched chain -- desirable -- carbon numbers 9-17 -- especially -- the alkyl group of carbon numbers 9-11 -- it is a straight chain alkyl group further especially. Although X shows hydrogen or a methyl group, it is hydrogen preferably. Although Y shows the alkylene machine of the straight chain of carbon numbers 1-5, or branched chain, it is an ethylene group preferably.

[0010] Things desirable in the fatty acid alkanolamide expressed with this general formula (1) are the fatty acid monoethanolamide expressed with the following general formula (2) or (3) or fatty acid diethanolamide, and monoethanolamide especially expressed with a general formula (2) preferably.

[0011] RCONHCH2CH2OH (2)

RCON(CH2CH2OH)2 (3)

(The inside of a formula and R show an above meaning.)

It can obtain them by the ability of the fatty acid alkanolamide expressed with the general formula (1) used in this invention to make the saturation of the straight chain of carbon numbers 8-22, or branched chain or unsaturated fatty acid, its low-grade ARUKIRU ester or glycerin ester, and alkanol amine able to react by a well-known method.

[0012] As an example of the fatty acid of the straight chain of carbon numbers 8-22, or branched chain used here Caprylic acid, capric acid, lauric acid, myristic acid, pulmitic acid, Stearic acid, oleic acid, coconut oil fatty acid, beef tallow fatty acid, palm oil fatty acid, Higher fatty acid, such as palm-kernel-oil fatty acid and synthetic fatty acid further obtained by paraffine oxidization or the OKISO method, etc. is mentioned, and the MECHIRU ester of these fatty acid, ethyl ester, etc. are mentioned as low-grade ARUKIRU ester of fatty acid.

[0013] Moreover, it is the compound which has an amino group with active hydrogen, and a hydroxyalkyl machine as alkanol amine. Mono-ethanol amine, JIETANORU amine, MONOISO propanolamine, JIISO propanolamine, methylethanol amine, MECHIRU iso propanolamine, etc. are specifically used, and they are mono-ethanol amine and JIETANORU amine

preferably.

[0014] The polyoxyalkylene fatty acid amide of this invention is obtained by making the fatty acid alkanolamide expressed with the general formula (1) obtained by the above methods, and the alkylene oxide of carbon numbers 2-4 react.

[0015] as alkylene oxide used by this invention, ethylene oxide, propylene oxide, butylene oxide, etc. are mentioned -- desirable -- ethylene oxide and propylene oxide -- it is ethylene oxide still more preferably.

[0016] [the alkylene oxide addition reaction in this invention] receiving fatty acid alkanolamide in basic catalysts, such as sodium hydroxide, water oxidization potassium, and sodium MECHIRATO, -- 0.01-5mol % -- [a thing / using is desirable and] When compounding fatty acid alkanolamide and using these basic catalysts, you may perform an alkylene oxide addition reaction as it is.

[0017] [in order it is satisfactory in this invention if alkylene oxide addition temperature is more than the melting point of the fatty acid alkanolamide expressed with a general formula (1), but to control the suboutput of thermal cracking, as much as possible, low temperature is good and changes also with kinds of alkylene oxide, but] When alkylene oxide is ethylene oxide, specifically, it is . More than the melting point of fatty acid alkanolamide is 80-100 degrees C more preferably below 110 degrees C. Although the number of addition Mol of alkylene oxide does not have restriction in particular, 1-20mol has the desirable number of average addition Mol of alkylene oxide, and 1-10mol is still more desirable. Thus, rough polyoxyalkylene fatty acid amide is obtained.

[0018] In addition, although the above-mentioned basic catalyst is included in this rough polyoxyalkylene fatty acid amide, before removal or neutralization of this basic catalyst performs the evaporation removal process of a low-boiling point ingredient with the following moisture, you may carry out by next any, but it carries out preferably later. Acetic acid, lactic acid, citrate, phosphorus acid, sulfuric acid, etc. are usually used for neutralization of a basic catalyst, and various adsorbent processings are effective in removal of a basic catalyst.

[0019] Next, although evaporation removal of a low-boiling point ingredient is performed with moisture from the rough polyoxyalkylene fatty acid amide obtained by doing in this way, if the above-mentioned purpose can be attained as this method, which method may be used, but the method shown in Following a and b is mentioned, for example. Among this, the method of a is more desirable than removal of a low-boiling point ingredient is efficiently possible.

[0020] a) How to perform by introducing steam or water the bottom of the condition more than **** of water, and in the system of reaction.

[0021] b) How to perform by making the inside of the system of reaction after introducing water in the system of reaction into the conditions more than **** of water.

[0022] In addition, it is desirable to adjust so that the quantity of a fraction, the steam to

introduce, or water may become almost the same in operation of a. Moreover, the water of the almost same weight as the weight removed by evaporation about b may be added again, and evaporation operation may be repeated further. removal operation of this low-boiling point ingredient -- a time part type and a continuation type -- efficiency improves and is desirable, if you may carry out by any and thin film-type evaporation equipment is used for a lot of factory production in that case.

[0023] It is desirable to carry out under decompression and these processes are 10 - 500mmHg, and further 20 - 200mmHg in the burden to equipment, or the field of efficiency especially. It is desirable to carry out. Moreover, in order to control the field of efficiency, degradation of hue, and generating of a nasty smell, 30-95 degrees C of temperature are desirable, and its 60-90 degrees C are especially desirable. Moreover, you may carry out, blowing inactive gas, such as nitrogen.

[0024] The quantity of the steam to introduce or water is 5 to 20 weight % preferably one to 50weight % to polyoxyethylene fatty acid amide. Productivity is also high by considering it as this range, and improvement of preservation stability can be performed efficiently.

[0025] Although the operation mechanism of this invention is not necessarily clear, it thinks as follows. namely, as a gelling mechanism under preservation of rough polyoxyalkylene fatty acid amide The aldehyde object which carries out subraw by the oxidization of an end hydroxyl group or the cleavage of an alkylene oxide chain produced in an alkylene oxide addition process condenses during preservation, and forms a structure, and it is possible to gel, and [with the method of this invention] Since the aldehyde object leading to gelling was removed efficiently, it is thought that gelling does not arise. It seems that this aldehyde object seemingly also caused muddiness and coloring and has also prevented muddiness and coloring simultaneously.

[0026]

[Example] Hereafter, although a work example explains this invention concretely, this invention is not limited to these. In addition, % in an example is a weight standard unless it mentions specially.

[0027] Work-example 1 lauric acid and mono-ethanol amine (molar ratio 1:1.01) It is made to react at 160 degrees C. Obtain lauric acid monoethanolamide and, subsequently NaOH 0.1 mol % is added. It is 2mol [per 1mol of lauric acid monoethanolamide] ethylene oxide at 100 degrees C 0.5-4kg/cm2 Polyoxy ethylene lauric acid amide (2mol of ethylene oxide addition) 200g obtained by having made react by pressure It teaches 500ml 4 mouth flask. While blowing steam over 2 hours by 80-90 degrees C and 50mmHg Fractions were collected. The quantity of the blown steam was 20g. Quantity of the steam blown at this time and the collected fractions was made the same.

[0028] It is sodium MECHIRATO (molar ratio 1:1.01:0.02) about work-example 2 lauric-acid

MECHIRU and mono-ethanol amine. It is made to react at 90 degrees C under existence. Lauric acid monoethanolamide is obtained. It ranks second. It is 2mol [per 1mol of lauric acid monoethanolamide] ethylene oxide at 100 degrees C. 0.5-4kg/cm2 [the polyoxy ethylene lauric acid amide (2mol of ethylene oxide addition) 200g obtained by having made react by pressure] While it teaches 500ml 4 mouth flask and water is dropped over 2 hours by 80-90 degrees C and 50mmHg Fractions were collected. The quantity of the added water was 20g. Quantity of the water added at this time and the collected fractions was made the same.

[0029] It compounds like the method of work-example 3 work-example 1 description from lauric acid myristic acid mixture fatty acid monoethanolamide (lauric acid/myristic acid = 65/35). 200g of obtained polyoxyethylene fatty acid amide (5mol of ethylene oxide addition) It taught 500ml 4 mouth flask, and fractions were collected, blowing steam over 2 hours by 80-90 degrees C and 50mmHg. The quantity of the blown steam was 20g. Quantity of the steam blown at this time and the collected fractions was made the same.

[0030] It compounds from palm-oil-fatty-acid monoethanolamide like the method of work-example 4 work-example 2 description. Obtained polyoxy ethylene palm-oil-fatty-acid amide (5mol of ethylene oxide addition) 200g It taught 500ml 4 mouth flask, and fractions were collected, blowing steam over 2 hours by 80-90 degrees C and 50mmHg. The quantity of the blown steam was 20g. Quantity of the steam blown at this time and the collected fractions was made the same.

[0031] It compounds from lauric acid diethanolamide like the method of work-example 5 work-example 1 description. Obtained bis(polyoxy ethylene) lauric acid amide (5mol of ethylene oxide addition) 200g It taught 500ml 4 mouth flask, and fractions were collected, blowing steam over 2 hours by 80-90 degrees C and 50mmHg. The quantity of the blown steam was 20g. Quantity of the steam blown at this time and the collected fractions was made the same.

[0032] It compounds from lauric acid diethanolamide like the method of work-example 6 work-example 1 description. Obtained bis(polyoxy ethylene) lauric acid amide (5mol of ethylene oxide addition) 200g It taught 500ml 4 mouth flask, and fractions were collected while water was dropped over 2 hours by 80-90 degrees C and 50mmHg. The quantity of the added water was 20g. Quantity of the water added at this time and the collected fractions was made the same.

[0033] Instead of blowing Steam 20g, except blowing Steam 4g, same operation was performed and polyoxy ethylene lauric acid amide was obtained in the work-example 7 work example 1.

[0034] In the comparative example 1 work example 1, except not performing steam processing, the same operation as a work example 1 was performed, and polyoxy ethylene lauric acid amide was obtained.

[0035] In the comparative example 2 work example 1, steam was not blown, but except

heating by 80-90 degrees C and 50mmHg for 2 hours, same operation was performed and polyoxy ethylene lauric acid amide was obtained.

[0036] The following method estimated the preservation stability of the polyoxyethylene fatty acid amide obtained by the example work examples 1-7 of an examination, and comparative examples 1-2, and the result was collectively shown in Table 1.

[0037] The hue and appearance at 50 degrees C when covering a <valuation method> 500ml sample bottle after N2 substitution and with a lid, and saving for 20 days at a 50-degree-C homoiothermy room are evaluated.

[0038]

[Table 1]

	保存後の色相	保存後の外観
実施例 1	G 1 ～ 2	透明液体
実施例 2	G 1 ～ 2	透明液体
実施例 3	G 1 ～ 2	透明液体
実施例 4	G 2	透明液体
実施例 5	G 1 ～ 2	透明液体
実施例 6	G 1 ～ 2	透明液体
実施例 7	G 2 ～ 3	ほぼ透明でゲル化もほぼなし
比較例 1	G 4	ゲル化
比較例 2	G 4	ゲル化

[Translation done.]